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<b>(21) International Application Number:</b> PCT/US94/14080 <b>(22) International Filing Date:</b> 7 December 1994 (07.12.94)  <b>(30) Priority Data:</b> 08/163,163 7 December 1993 (07.12.93) US  <b>(71) Applicant:</b> THE NUTRASWEET COMPANY [US/US]; P.O. Box 730, 1751 Lake Cook Road, Deerfield, IL 60015-5239 (US).  <b>(72) Inventors:</b> TSAU, Josef; 15358 West Fair Lane, Libertyville, IL 60048 (US). DESAI, Nitin; 1012 North Meadow Lane, Mt. Prospect, IL 60056 (US). BRAY, Ronald, G.; 1303 Whitney Lane, Buffalo Grove, IL 60089 (US).  <b>(74) Agents:</b> BELL, Craig, M. et al.; Paul & Paul, 2900 Two Thousand Market Street, Philadelphia, PA 19103 (US).		<b>(81) Designated States:</b> AU, CA, FI, JP, KR, NO, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>  <b>BEST AVAILABLE COPY</b>
<b>(54) Title:</b> STABLE AQUEOUS DIPEPTIDE SUSPENSIONS		
<b>(57) Abstract</b>		
<p>A shelf stable, aqueous suspension of a high intensity dipeptide sweetener such as aspartame is made possible through the incorporation of trace amounts of a food grade stabilizing flow agent. The stabilizing flow agent is selected from the group consisting of food grade polymers, hydrocolloids, gums and mixtures thereof. The suspension can comprise high levels of aspartame that is of a viscous consistency and highly soluble, yet remains pourable and easy to handle.</p>		

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Stable Aqueous Dipeptide SuspensionsBackground of the Invention

The present invention relates to the preparation of stable suspensions of high intensity sweeteners for use in foods and beverages and the compositions prepared thereby. More specifically, the present invention relates to stable dipeptide based sweeteners that are formulated as stable, easy to use suspensions for use in the food and beverage industries.

Aspartame (APM) is a well known dipeptide sweetener manufactured and sold by The NutraSweet Company of Deerfield, Illinois, as NutraSweet Brand sweetener. Additionally, there are some foreign manufacturers of aspartame. When used herein the term "aspartame" or "APM" refers to alpha-L-aspartyl-L-phenylalanine methyl ester and its' salts and metal complexes as taught in U.S. Patent No. 3,492,131 to Schlatter. Salts and metal complexes of APM are disclosed in U.S. patents 4,439,460; 4,029,701; 3,714,139; 4,031,258; and 4,448,716 all of which are incorporated herein by reference.

A major application for aspartame is as a low calorie sweetener in the food and beverage industries. Aspartame is sold in bulk to the various beverage manufacturers who must then repackage the sweetener and distribute it to their various bottlers. Aspartame in bulk is commercially available as both powder and granulated forms. Aspartame powder has low bulk density and good dissolution properties yet, due to its lower bulk density and small particle size, is readily air-borne and may have dusting and flow problems. The granular form has a higher bulk density and possesses good flow properties but does not always

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instantaneously dissolve. These properties can result in product losses and metering problems, a need for expensive packaging and overall shipping and handling inconveniences.

5       The present invention improves the overall stability and flowability of aspartame during shipping and storage. The dipeptide nature of the compound, render it highly susceptible to degradation by heat and other chemicals by hydrolysis of the its peptide bonds.  
10 Long term storage or storage in areas of high temperatures often result in the loss of much of its sweetness. Moreover, the powder itself is hard to handle as some becomes lost in the environment as "dust". It has been determined that in the beverage  
15 industry some of the bulk aspartame powder is lost as dust in the air during handling and manufacture. When one is dealing in tons of aspartame per year, these are considerable losses which if cut down will result in major cost reductions. The present invention  
20 eliminates this problem.

Finally, another advantage of the stabilized APM suspensions of the present invention is the dissolution and dispersion of the suspensions in solution as compared with the unprocessed aspartame powder. During  
25 beverage manufacture, aspartame powder when mixed with the soft drink liquid has a tendency to float on the surface of the liquid and adhere to the sides of the container in which it is mixed. In non-acidified liquids, both the powder and granular forms tend to  
30 clump into a sticky mass and are not easily dispersed and dissolved. Since the high tech beverage manufacturing facilities of today require precise metering of the liquid components of the beverage to be made, such clumping is entirely unacceptable. This  
35 requires the use of equipment for agitation or stirring

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to mix the powder into the liquid resulting in additional processing steps and costs of manufacture.

For liquid and semi-liquid applications a high concentration aspartame (APM) suspension can have many advantages over powder or granular APM. For example, it may be more convenient and economical for carbonated soft drink companies to have an all liquid production system to avoid the costs, labor and problems relating to handling solids, particularly in powder form. However, both APM's aqueous stability and solubility have prevented the development of such a product.

United States Patent No. 4,722,844 to T. Ozawa et. al. teaches the preparation of aqueous APM suspensions which are asserted to be stable both chemically and physically. In water, like most crystalline materials, APM particles sink and settle since APM's specific gravity is significantly greater than that of water. To obtain physically stable APM suspensions, this patent teaches the addition of a viscosity or specific gravity increasing component, such as food gums and polysaccharides. The patent discloses physically stable 2-5% APM suspensions in sugar syrups, such as isomerized sugar and sorbitol syrups. The aspartame suspensions of Ozawa et. al. '844 use an aqueous vehicle that is high in sugar, polysaccharide or food gum content, i.e. over 50%, and low aspartame content. Such a product will not be acceptable however, for use in most food applications.

United States Patent No. 4,007, 288 to Glicksman et. al. discloses a readily soluble sweetening composition for use in foods and beverages wherein the aspartame is first solubilized with a bulking agent such as dextran or some other hydrolyzed starch material and the solution is then vacuum drum dried to a composite powder of sweetener and bulking agent.

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Brief Description of the Drawings

Figure I is a line graph plotting the percent of dissolved aspartame with a trace amount of carboxymethyl cellulose in water as a function of time.

5        Figure II is a line graph comparing the dissolution rate of the aspartame suspensions of the present invention versus that of pure aspartame alone in water

10       Figure III is a graph depicting the effect of the hydrocolloid additives on the viscosity of APM suspensions.

Figure IV is a flow curve of aspartame suspensions using sodium carboxymethyl cellulose.

15       Figure V is a graph depicting the fluidization of APM suspensions using a hydrocolloid.

Detailed Description of the Invention

For liquid and semi-liquid applications, a high concentration aspartame (APM) solution can have many advantages over powder and granular APM as discussed, supra. For example, it may be more convenient and economical for carbonated soft drink companies to have an all liquid production system to avoid the costs, labor and problems relating to handling solids. However, both APM's aqueous stability and solubility characteristics have prevented the development of such a product.

20       25

The present invention is a relatively pure aspartame liquid suspension with an APM concentration of from about 10% to 70% by weight that is both physically stable under long term and adverse storage conditions as well as providing greater ease in handling than bulk aspartame crystalline powder. The problem with most APM suspensions of high aspartame concentrations, i.e. >20%, is that they become highly

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viscous, sticky and not sufficiently flowable for the required applications.

By the term "suspension" one refers to a system whereby very small particles (solid, semi-solid or liquid) are more or less uniformly dispersed in a liquid or gaseous medium. If the particles are small enough to pass through a filter membrane the system is colloidal. If the particles are larger than this, they will tend to precipitate and sink since they are heavier than the surrounding solution. If lighter than the solution however, they will agglomerate and rise to the surface.

All of the aforementioned problems are surprisingly overcome in the present invention through the incorporation of a small amount of a stabilizing flow agent comprising a food grade polymer, a hydrocolloid or gum such as sodium carboxymethyl cellulose, algin, gum arabic, carrageenan, xanthan gum, guar gum, hydroxypropyl methyl cellulose (HPMC), methylcellulose, pectin, locust bean gum, sodium alginate, propylene glycol alginate, caramel and mixtures thereof. Trace amount of an emulsifier or wetting agent such as polysorbate (polyoxyethylene fatty acid ester) or lecithin may also be incorporated to improve the suspensions' dissolution and stability characteristics. The food polymers, emulsifiers, hydrocolloids or gums may be effectively incorporated in the suspensions in amounts of from about 0.001% to about 0.5% of the total weight of the suspension. Levels exceeding 0.5% may continue to reduce the viscosity of the suspension but to a less effective degree and thereby provides a diminished return.

Although aspartame powder is sparingly soluble in water, it is possible to make a suspension of APM in water by itself. For example, suspensions up to 30%

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can be made in water, however, this suspension is a very viscous foam or paste which does not lend itself to fluid processing operations like pumping, mixing, etc., which are prevalent in the beverage and dairy industries. This foam is not stable and separates into 3 phases, a foam/air layer, water and a layer of APM crystals that has settled. The addition of the stabilizing flowability agent of the present invention in a trace amount (approx. 0.2%) during the formation of the suspension makes it stable which lends itself well to unit operations in fluid processing for the food and beverage industry.

The addition of the food grade polymers, hydrocolloids or gums to an aqueous suspension of aspartame surprisingly alters the flow characteristics of the suspension to a more fluid form exhibiting pseudoplastic flow behavior with enhanced dissolution properties and stability. This is of particular value to applications involving fountain dispensed carbonated soft drinks where aspartame is prone to accelerated degradation because of the low pH environment created by the other fountain components.

When mixed in a suspension with the stabilizing flow agent, aspartame levels of from approximately 10% to about 70% by weight of the total weight of the suspension is achievable. Preferably, said aspartame is incorporated in amounts of from about 20% to about 55% by weight and most preferably in an amount of from about 20% to about 35% by weight of the entire suspension.

Viscosity values for suspensions can be measured using a Haake Rotoviscometer Model VT500 (Saddlebrook, N.J.) in which a 'bob' rotates in a vessel containing the suspension to be measured. As the bob rotates, the speed is measured as the shear rate and the

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suspension is sheared and exerts a resistance to the movement of the bob that is measured by the stress which is a direct function of its viscosity; i.e. the greater the viscosity, the greater the resistance.

5 This is the shear stress plotted on the Y-axis of Figures III-V Viscosity (ETA) is measured by the slope of the line plotted by the shear stress v. shear rate. This is called the flow curve and the viscosity values are defined in terms of milli-Pascal seconds (mPas).

10 Referring now to the graph of Figure I, it is clearly shown just how quickly the compositions of the present invention dissolve in water. A 50% APM suspension with 0.2% carboxymethyl cellulose is almost completely dissolved within thirty (30) seconds.

15 Figure II compares the rate of dissolution of the 50% APM suspension (with 0.2% CMC) versus that of pure APM powder in distilled water at 10°C. Again clearly, a greater percentage of APM becomes dissolved in solution at a much faster rate using the compositions of the present invention than that of APM alone.

20 Traditionally, hydrocolloids have been used to increase the viscosity of fluids. In the present case, the addition of hydrocolloids turns the foam into fluid, the viscosity decreases, and the APM suspension becomes fluidized thereby becoming more manageable.

25 Microscopy data shows that the APM crystals surround the air bubbles in a foam and that crystals are floating or moving freely in a liquid suspension. This phenomena may be explained by the fact that APM is

30 dipeptide with a hydrophobic group in its structure. When this is dissolved in water, the water is organized and further addition of the crystals then creates the foam with the aspartame stabilizing the foam. Addition of a very hydrophilic polymer like carrageenan or

35 sodium carboxymethyl cellulose (sodium CMC) frees up

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the water and allows more APM to be added to the system. The suspending properties of sodium CMC are not unknown, however, the fluidizing effect it has on the APM suspension is truly surprising and unexpected.

5 Referring now to Figure III, the effect of some selected hydrocolloids on the viscosity of APM suspensions is compared to a control 30% solution of pure aspartame. ETA, i.e. viscosity of the suspension, is plotted as a function versus the shear rate of the  
10 suspensions. The greater the slope of the line, the greater the breakdown and flowability of the system. As can be seen from the graph, suspensions comprising 30% aspartame with 0.2% sodium carboxymethyl cellulose or 0.2% Viscerin (carrageenan) maintain viscosities  
15 that break down with the application of greater shear than that of the 30% APM control. Therefore, the suspensions with the additional hydrocolloid excipient were of a lower viscosity than the control indicating the flowability of these suspensions is also far  
20 greater.

Figure IV on the other hand shows the flow curve of three APM-CMC suspensions at 25°C through a comparison of the compositions' shear stress as plotted against its shear rate. Again, flowability is measured  
25 in terms of the slope of the different curves and as expected, the 30% APM suspension displays far quicker breakdown at a greater rate than the 40% APM suspension which is greater than that of the 50% APM suspension. Nevertheless, the 50% APM-CMC suspension does display  
30 substantial breakdown with increasing shear rate thereby proving that even high APM concentrations of the present invention are flowable and pourable and useful in the fluid and semi-fluid applications of the food and beverage industry.

35 Finally, Figure V compares the fluidability of a

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control 30% APM suspension with that of two 30% suspensions of the present invention comprising APM with trace amounts of two different hydrocolloids. Again, the rate of shear stress, i.e., the viscosity, is far greater with respect to the control than the two suspensions of the present invention. Here, the shear exerted against the system is encountering far greater resistance from the control (as shown by the height of the control graph line) than the other two suspensions. Hence, the compositions of the present invention are far more fluid and consistent in their texture.

Without being bound to any theory, it is believed that the addition of the hydrocolloids, gums, or food grade polymers prevents the formation of foam, i.e., the air entrapment by APM crystals and forms a fluid suspension of APM crystals in water with little or no air entrapment. In light micrographs of the foam APM in water, the crystals surround the air bubbles whereas in the suspension made with hydrocolloids/gums/food grade polymers, the crystals are literally moving around in the continuous water phase.

The present invention also considers the method of preparing the physically stable and flowable APM suspensions. The desired amount of aspartame for a given concentration in either powder or granular form may be dispersed in water either with or without the food grade polymer flow agent using a standard intense rotary mixer. The preferred aspartame for use is a powder form of crystalline particles with a size of no greater than 100 standard U.S. mesh. Aspartame granules and purified wet aspartame precipitate formed as a wet cake may also be used, provided they are mixed well into solution. The flow agent may be mixed into the aspartame powder or precipitate prior to solubilization or may be added to all three types once

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in solution.

Aspartame powder or granules are added slowly to water in a kettle and mixed using a high shear mixer in order to break up the larger particles. The  
5 stabilizing flow agent such as sodium CMC is added along with the powder. This helps in reducing the viscosity and allows for the addition of more APM. If preferred, sodium CMC can also be added to the water first and thoroughly dispersed prior to the subsequent  
10 addition of the APM to make the suspension.

The greater the concentration of aspartame in the suspension, the greater the viscosity of the suspension. Obviously, suspensions of long term stability will be more highly viscous than those of  
15 short term stability. Generally, the preferred viscosity values for the suspensions of the present inventions should range from about 150 mPas. to about 1750 mPas. and most preferably from about 150 mPas. to about 750 mPas. at 50 l/s shear rate when measured in  
20 the Haake Roto-viscometer at 25°C.

The flowable APM suspensions of the present invention may have many applications and can replace powder and granular APM in all liquid or semi-liquid food uses. In particular, the suspensions will provide  
25 a shelf stable product that is easily handled and readily incorporated into soft drinks, juices, dairy products and other beverage applications. Its high rate of dissolution and chemical stability will be particularly beneficial in fountain dispensed soft  
30 drink applications. It can also be used as an excellent table top sweetener with its superior dust free, rapid dispersion and dissolution properties.

The fluid APM suspensions of the present invention are an excellent sweetener source in "high tech"  
35 continuous beverage manufacturing plants employing

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computer controlled valves that require precise delivery and mixing of liquid ingredients or components of the beverage. The APM suspension mixes easily with water or other sweetening agents such as high fructose corn syrup, liquid sucrose and/or fructose with minimal agitation or mixing and can be used in the manufacture of diet beverages in combination with caloric sweeteners.

The following examples are provided to better describe and disclose several embodiments of the present invention. They are for illustrative purposes only however, and it is recognized that minor variations and changes may be made with respect to the ingredients or process parameters that differ from those set forth below. These are still considered to fall within the spirit and scope of the present invention however, as recited by the claims that follow.

#### Example I

A number of APM suspensions at 30% concentration were prepared using the stabilizing flow agents of the present invention comprising the following food polymers, hydrocolloids and gums in an amount of approximately 0.2% by weight of the total weight of the suspension. The flow curves of the suspensions were generated using the Haake Rotoviscometer Model VT500 at 25°C using a MV1 sensor. The viscosity at 54 1/s is reported.

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		Viscosity at
<u>Polymer Flow Agent</u>		<u>54 (1/s) shear rate 25°C</u>
	Carrageenan	32
	Sodium Alginate	88
5	Xanthan gum	63
	Sodium carboxymethyl cellulose	111
	Locust bean gum	519
	Guar gum	193
	Pectin	1512
10	Gum Arabic	679
	Propylene glycol alginate	18
	Caramel DS #400	166

A combination of polymer flow agents can also be used to make fluid 30% APM suspensions in water as shown below:

		Viscosity at
<u>Polymer Flow Agent</u>		<u>54 (1/s) shear rate 25°C</u>
	0.2% Carrageenan + 0.05% Xanthan	58
20	0.2% Carrageenan + 0.05% Locust bean gum	68
	0.2% Carrageenan + Guar gum	43
	0.2% Carrageenan + 0.05% Sodium carboxymethyl cellulose	64

Samples of the above suspensions (50 ml.) were placed in test tubes and spun in a table-top centrifuge for fifteen (15) minutes at 50 g. After this time, all suspensions exhibited less than 2.0% sedimentation in the tubes.

#### Example II

Stable APM suspensions were incorporated as the sweetener in fountain dispensed diet beverages and carbonated soft drinks (CSD).

Diet concentrates for fountain dispensed beverages need a stable high intensity sweetener since the distribution of the concentrate takes an average of 75 days in distribution before it is hooked to a dispensing unit. The pH of the concentrate is below 3.0. APM degradation in such applications is well known in the beverage industry. There are two modes of delivering the beverage concentrate to the fountain

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unit:

1. A bag or a box container, typical size 5 gallons.
2. A pressurized cylinder.

5 The preparation of a stable suspension with enhanced dissolution characteristics makes it possible to use APM in fountain dispensed drink applications.

Stable APM suspensions can be used in such applications where the suspension is stored separately  
10 from the diet drink/beverage concentrate in a separate unit. A required amount of the suspension (approx. 110 ml. of 50% APM for 5 gallon concentration) can be injected into the concentrate prior to attachment to the fountain dispenser. The injected APM dissolves  
15 rapidly in the concentrate within 30 seconds of introduction in the container under quiescent conditions with minimal external agitation providing the necessary sweetness when the beverage is dispensed from the fountain unit.

20 Food grade emulsifiers or wetting agents such as polysorbate 60 or lecithin may be added to the suspension to further enhance the dissolution and stability.

#### Example III

25 A 50% liquid APM suspension containing 0.2% CMC was spray dried in a Niro spray drier with a centrifugal head. The inlet temperature was 160°C and the outlet temperature was 100°C. The resulting powder was then easily reconstituted into a suspension. The  
30 powder can also be agglomerated using standard spray drying and agglomeration technology. The wet cake (described earlier) can be fluidized using the polymer flow agent and spray dried into a powder.

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What We Claim is:

1. A shelf stable, aqueous sweetener suspension comprising a high intensity dipeptide sweetening agent and a stabilizing flow agent.
- 5        2. The shelf stable, aqueous sweetener suspension of claim 1 wherein said high intensity dipeptide sweetener is alpha-L-aspartyl-L-phenylalanine methyl ester, its salts and metal complexes thereof.
- 10       3. The shelf stable sweetener suspension of claim 1 wherein said food grade stabilizing flow agent is selected from the group consisting of polymers, hydrocolloids, gums and mixtures thereof.
- 15       4. The shelf stable suspension of claim 3 wherein said stabilizing polymer flow agent is selected from the group consisting of sodium carboxymethyl cellulose, dextran, algin, gum arabic, carrageenan, xanthan gum, guar gum, hydroxy-propylmethyl cellulose, methyl cellulose, pectin, locust bean gum, sodium alginate, propylene glycol alginate, caramel and  
20       mixtures thereof.
5. The shelf stable sweetener suspension of claim 4 wherein said aspartame is present in said suspension in an amount of from about 10.0% to about 70% by weight of the total weight of the suspension.
- 25       6. The shelf stable sweetener suspension of claim 5 wherein said aspartame is present in an amount of from about 20% to about 55% by weight of the total weight of the suspension.
7. The shelf stable sweetener suspension of

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claim 6 wherein said aspartame is present in an amount of from about 20% to about 35% by weight of the total weight of the suspension.

8. The shelf stable sweetener suspension of  
5 claim 7 wherein said food grade polymer flow agent is present in an amount of from about 0.001% to about 0.6% by weight of the total weight of the suspension.

9. The shelf stable sweetener suspension of  
10 claim 8 wherein said food grade polymer flow agent is present in an amount of from about 0.1% to about 0.3% by weight of the total weight of the suspension.

10. The shelf stable sweetener suspension of claim 9 further comprising an emulsifier.

11. The shelf stable sweetener suspension of  
15 claim 10 wherein said emulsifier is selected from the group consisting of polysorbate, lecithin and mixtures thereof.

12. The shelf stable sweetener suspension of  
20 claim 11 wherein said aspartame is comprised of aspartame particles no greater than 100 standard U.S. mesh.

13. The shelf stable sweetener suspension of claim 12 wherein said aspartame concentration renders said suspension it highly viscous.

25 14. The shelf stable sweetener suspension of claim 13 wherein said viscosity ranges from about 150 mPas. to about 1750 mPas.

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15. The shelf stable sweetener suspension of claim 14 wherein said viscosity ranges from about 150 mPas. to about 750 mPas.

16. A method for the preparation of a shelf  
5 stable, aqueous dipeptide sweetener comprising blending a powdered or granular aspartame composition with a stabilizing flow agent and solubilizing said mixture to a desired concentration in water.

17. The method of claim 16 wherein said  
10 stabilizing flow agent is selected from the group consisting of food grade polymers, hydrocolloids, gums and mixtures thereof.

18. The method of claim 17 wherein said  
stabilizing flow agent is selected from the group  
15 consisting of sodium carboxymethyl cellulose, dextran, algin, gum arabic, xanthan gum, guar gum, hydroxypropyl methylcellulose, methyl cellulose, pectin, locust bean gum, carrageenan, caramel and mixtures thereof.

20 19. The method of claim 18 wherein said aspartame is present in an amount of from about 10.0% to about 70% by weight of the total weight of the suspension.

20. The method of claim 19 where said aspartame  
is present in amount of from 20% to about 55% by weight  
25 of the total weight of the suspension.

21. The method of claim 20 wherein said aspartame  
is present in an amount of from about 20% to about 35%  
by weight of the total weight of the suspension.

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22. The method of claim 21 wherein said stabilizing flow agent is present in an amount of from about 0.001% to about 0.6% by weight of the total weight of the entire suspension.

5        23. The method of claim 22 wherein said stabilizing flow agent is present in an amount of from about 0.1% to about 0.3% by weight of the total weight of the suspension.

10       24. The method of claim 23 further comprising an emulsifier.

25. The method of claim 24 wherein said emulsifier is selected from the group consisting of polysorbate, lecithin and mixtures thereof.

15       26. The method of claim 25 wherein said aspartame is first mixed into solution and said stabilizing flow agent and emulsifier are subsequently added.

20       27. A shelf-stable, highly viscous, highly soluble aqueous aspartame suspension useful as a sweetening agent in liquid and semi-liquid food applications.

28. The shelf-stable, aqueous aspartame suspension of claim 27 useful as a sweetener in carbonated soft drinks, fountain dispensed beverages, dairy products, 25 juices and other beverages.

29. The shelf stable aqueous aspartame suspension of claim 28 useful as a table top sweetener.

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30. An improved fountain dispensed beverage sweetener with a stable aqueous dipeptide sweetener suspension.

31. The fountain beverage of claim 30 wherein  
5 said aqueous sweetener suspension comprises aspartame and a stabilizing flow agent.

32. The fountain beverage of claim 31 wherein  
said stabilizing flow agent is selected from the group  
consisting of food grade polymers, hydrocolloids, gums  
10 and mixtures thereof.

33. The fountain beverage of claim 32 wherein  
said beverage is carbonated.

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Dissolution of Liquid APM (50% Suspension,  
0.2% CMC) in Distilled Water at 10 °C

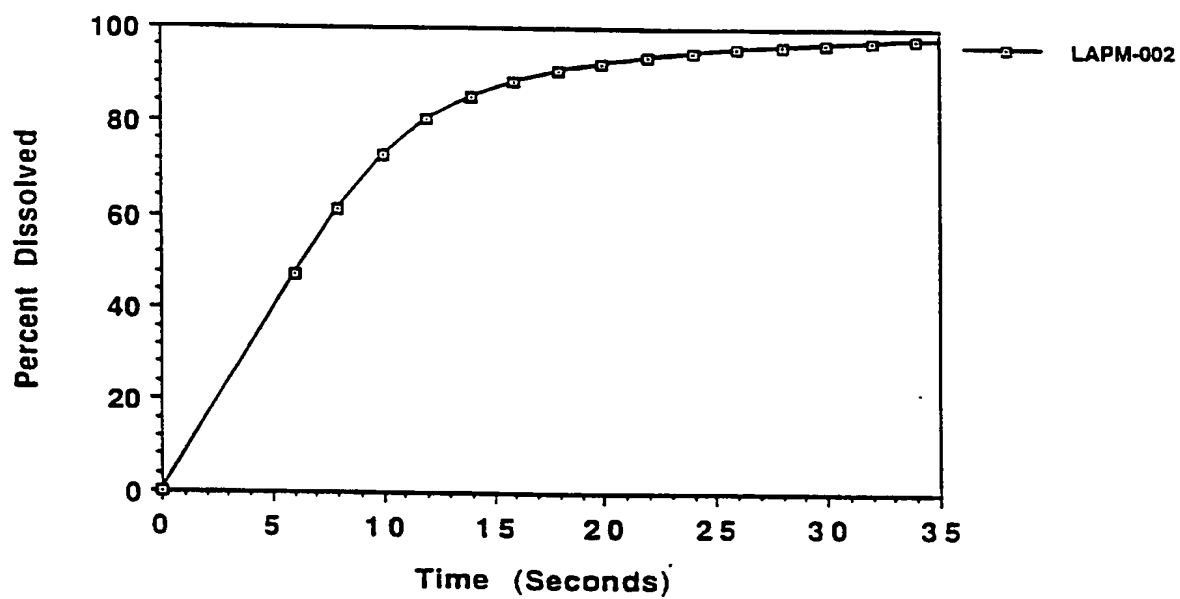


FIGURE I

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Comparison of Dissolution of Liquid APM  
(50% Suspension, 0.2% CMC) and APM  
Powder in Distilled Water at 10 °C

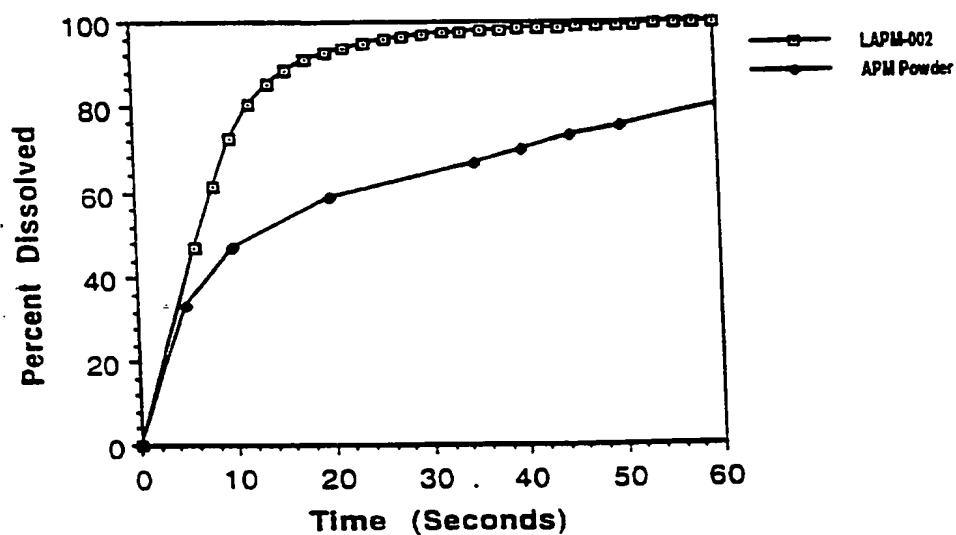


FIGURE II

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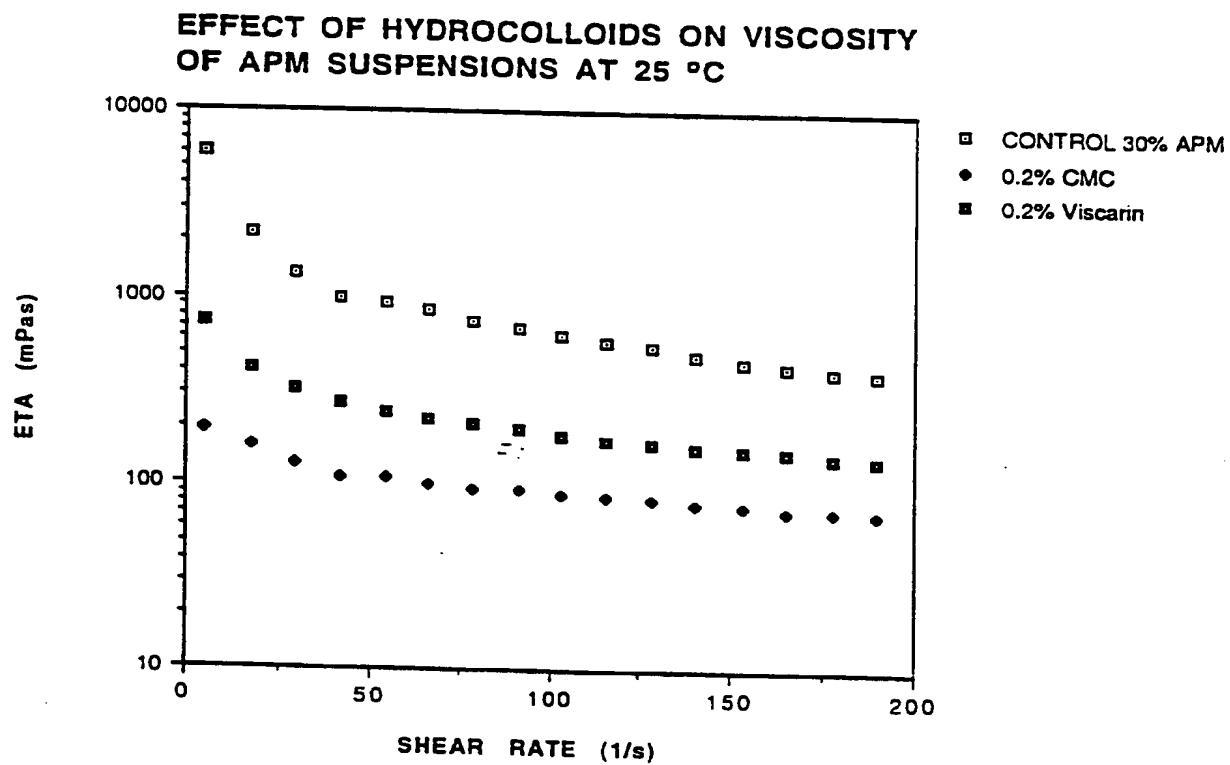


FIGURE III

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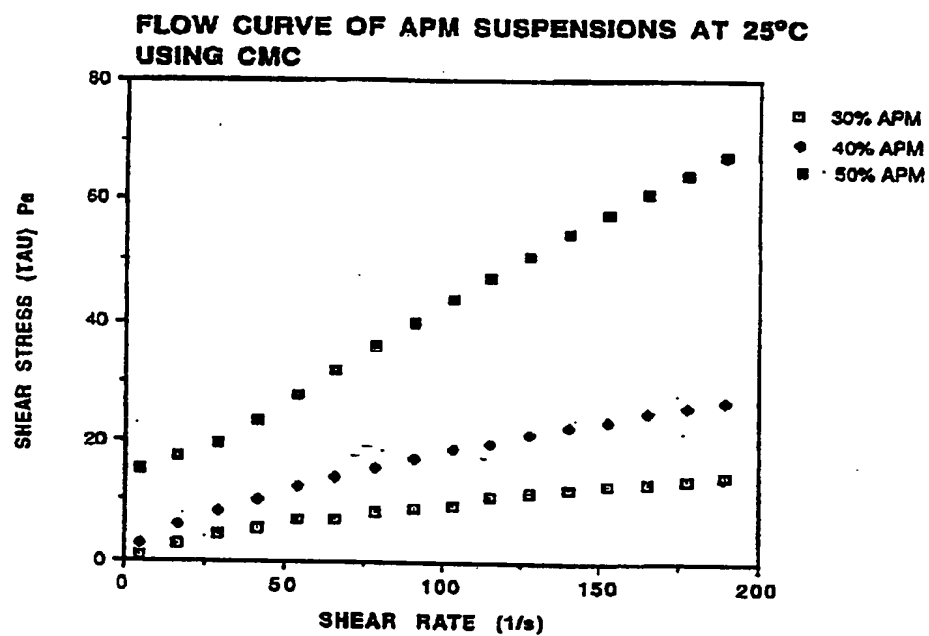


FIGURE IV

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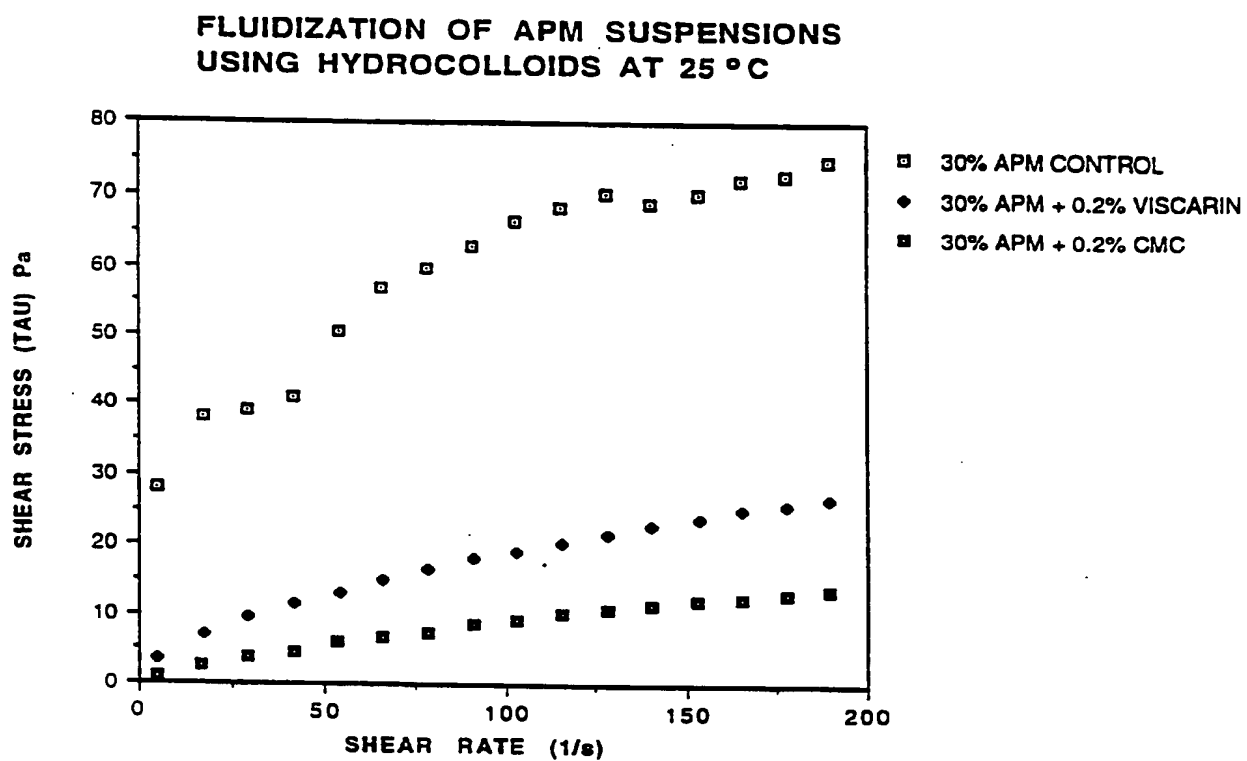


FIGURE V

## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US94/14080

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :A23L 1/236

US CL :426/548

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 426/548

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched  
noneElectronic data base consulted during the international search (name of data base and, where practicable, search terms used)  
none

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X --- Y	US, A, 4,722,844 (OZAWA ET AL.) 02 February 1988, See the Abstract; column 3, lines 49-63; column 4, lines 14-24.	1-4, 16-18, 27-33 ----- 5-15, 19-26

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